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METHODS FOR CHEMICAL ANALYSIS OF SILVER SOLDER

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF SILVER SOLDER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 October 1956, on approval by the Engineering Division Council of the draft finalized on 11 September 1956 by the Basic Non-Ferrous Metals Sectional Committee.

0.2 The methods of analysis prescribed in this standard have been prepared with a view that they shall be useful as reference methods. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.3 Taking into consideration the views of producers, consumers and testing authorities, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the technological methods followed in the country in this field. This consideration led the Sectional Committee to derive assistance from the 1950 Book of ASTM Methods for Chemical Analysis of Metals.

0.4 This standard is a necessary adjunct to Indian Standard Specification for Silver Solder (*IS : 192-1956). Besides, this standard requires reference to the following Indian Standard Specifications:

IS : 264-1950 NITRIC ACID

†IS : 266-1950 SULPHURIC ACID

†IS : 323-1952 RECTIFIED SPIRIT

0.4.1 Wherever a reference to any specification mentioned under **0.4** appears in this standard, it shall be taken as a reference to the latest version of the specification.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with †IS : 2-1949 Rules for Rounding Off Numerical Values.

*Superseded by IS : 2927-1964 Specification for Brazing Alloys.

1. SCOPE

1.1 This standard covers the methods of chemical analysis of various grades of silver solder. The methods described deal with the determination of silver, copper, cadmium, and zinc.

2. SAMPLING

2.1 Sample shall be drawn as specified under 6 of *IS: 192-1956.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals shall be employed in tests, and distilled water shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF SILVER

4.1 Reagents — The following reagents are required.

4.1.1 Dilute Nitric Acid — 1:4 (by volume).

4.1.2 Dilute Hydrochloric Acid — 3:100 and 1:500 (by volume).

4.2 Procedure — Dissolve 1 g of the material, accurately weighed, into 20 ml of dilute nitric acid, and boil to expel brown fumes. If any precipitate of metastannic acid is formed, filter it off through a close texture paper and wash with hot water. (It shall, however, be made sure before filtering that the precipitate was not due to contamination by chlorides from any source.) Dilute the solution to 150 ml with water, heat and add dilute hydrochloric acid (3:100) slowly and with constant stirring until precipitation is complete. Allow to settle away from direct light. Test the supernatant liquid with 1 or 2 drops of dilute hydrochloric acid (3:100) to make sure that precipitation is complete. If no turbidity appears, filter through a weighed porcelain Gooch crucible or sintered glass crucible and wash the precipitate several times with dilute hydrochloric acid (1:500). Dry the precipitate first at 90°C for 30 minutes and then at 180°C to constant weight. Cool in a desiccator and weigh as silver chloride (Ag Cl).

4.2.1 Reserve the filtrate for the determination of copper under 5.

4.3 Calculation

$$\text{Silver, percent by weight} = 75.26 \frac{A}{B}$$

where

A = weight in g of silver chloride, and

B = weight in g of the material taken for the test.

*Superseded by IS: 2927-1964.

5. DETERMINATION OF COPPER

5.1 Apparatus—The type of apparatus recommended is that with platinum electrodes with or without rotating agitator.

5.1.1 Cathode—The cathode shall be of platinum which may be formed either from sheet or wire gauze. It shall give a deposition surface of about 100 sq cm.

5.1.2 Anode—The anode shall be of either platinum solid spiral or wire gauze.

5.1.3 Beaker—a 250 ml tall lipless beaker with well fitting split cover-glasses.

5.2 Reagents—The following reagents are required.

5.2.1 Concentrated Sulphuric Acid—sp gr 1.84 (conforming to *IS:266-1950).

5.2.2 Concentrated Nitric Acid—sp gr 1.42 (conforming to *IS:264-1950).

5.2.3 Rectified Spirit—95 percent by volume (conforming to *IS:323-1952).

5.3 Procedure—To the filtrate reserved under 4.2.1, add 5 ml of sulphuric acid and evaporate to dense white fumes. Cool, dilute with 50 ml of water and boil. Transfer to the lipless beaker, add 2 ml of nitric acid and dilute to 100 ml with water. Insert the electrodes into the solution, cover with the split cover glasses and electrolyse with a current of 0.5 amperes without agitation for overnight, or with a current of 2 to 3 amperes, using gauze cathode and rotating agitator for about 2½ hours. When the solution is colourless, wash down the cover glasses, electrodes and sides of the beaker, raising the level of the liquid slightly. Continue the electrolysis until no copper is deposited on the newly exposed surface of the cathode. When the deposition is complete, continue the current and raise the cathode gradually as it is thoroughly washed with water, catching the washings in the beaker. Dip the cathode in two successive baths of rectified spirit, dry for 3 to 5 minutes at 105 to 110°C, cool in a desiccator and weigh the deposit as metallic copper.

5.3.1 Reserve the electrolyte for the determination of cadmium under 6, or zinc under 7.

5.4 Calculation

$$\text{Copper, percent by weight} = \frac{A}{B} \times 100$$

*Since revised.

where

A = weight in g of copper, and

B = weight in g of the material taken for the test.

6. DETERMINATION OF CADMIUM

6.1 Reagents — The following reagents are required.

6.1.1 Dilute Ammonium Hydroxide Solution — 1:1 (by volume).

6.1.2 Dilute Sulphuric Acid — 1:1 and 1:4 (by volume).

6.1.3 Hydrogen Sulphide — gas.

6.1.4 Dilute Hydrochloric Acid — 1:3 (by volume).

6.1.5 Concentrated Sulphuric Acid — sp gr 1.84 (conforming to *IS:266-1950).

6.1.6 Ammonium Chloride — solid.

6.1.7 Concentrated Nitric Acid — sp gr 1.42 (conforming to *IS:264-1950).

6.2 Procedure — Heat the electrolyte reserved under 5.3.1 to boiling, make slightly alkaline with dilute ammonium hydroxide solution and then just neutral with dilute sulphuric acid (1:4). Add 25 ml of dilute sulphuric acid (1:4) in excess, dilute to 300 ml with water, bring to a boil, and pass hydrogen sulphide into the solution for one hour allowing it to cool. If no precipitate appears within a few minutes, add dilute ammonium hydroxide solution drop by drop, until precipitation starts. Filter through a close texture paper and wash twice with warm water. Transfer as much of the precipitate as possible to the beaker in which the precipitation was made and dissolve the precipitate in the beaker and on the paper in hot dilute hydrochloric acid. Wash the paper well with water. Add 10 ml of sulphuric acid (sp gr 1.84) to the solution and evaporate to white fumes. Cool, add 300 ml of water and 5 g of ammonium chloride. Bring the solution to a boil and pass hydrogen sulphide for one hour. Filter, wash and dissolve the precipitate and repeat the sulphide precipitation as described above. Filter, wash and dissolve the final precipitate of cadmium sulphide in dilute hydrochloric acid as before and evaporate the solution to low bulk. Transfer to a weighted platinum crucible, add 10 ml of dilute sulphuric acid (1:1) and evaporate to white fumes. If the solution darkens, cool, add a little water and 5 ml of nitric acid and again evaporate to white fumes. Remove the excess acid by heating carefully and increasing the heat to 500 to 600°C. Cool, dissolve the residue in a little water, evaporate carefully and heat again to 500 to 600°C. Cool in a desiccator and weigh as cadmium sulphate.

*Since revised.

6.2.1 Reserve the combined filtrates from the cadmium sulphide precipitates for the determination of zinc under 7.

6.3 Calculation

$$\text{Cadmium, percent weight} = 53.92 \times \frac{A}{B}$$

where

A = weight in g of cadmium sulphate, and

B = weight in g of the material taken for the test.

7. DETERMINATION OF ZINC

7.1 Reagents—The following reagents are required.

7.1.1 Ammonium Hydroxide Solution—sp gr 0.90.

7.1.2 Formic Acid—sp gr 1.20.

7.1.3 Hydrogen Sulphide—gas.

7.1.4 Formic Acid Wash Solution—Dilute 4 ml of formic acid to one litre with water and saturate with hydrogen sulphide.

7.1.5 Dilute Hydrochloric Acid—1 : 1.

7.1.6 Standard Potassium Ferrocyanide Solution—Dissolve 16.166 g potassium ferrocyanide and 0.3 g potassium ferricyanide in one litre of water. Standardize against pure zinc before use.

7.1.6.1 Standardization of the solution—Take 0.25 g of pure metallic zinc and dissolve in 25 ml of dilute sulphuric acid (1 : 4). After dissolution, neutralize the solution with ammonium hydroxide and then make it just acidic with dilute sulphuric acid (2 : 98) with the help of a litmus paper. Add 25 ml of dilute sulphuric acid (1 : 4). Just boil and cool to room temperature. Add 20 ml of 20 percent ammonium chloride solution and 3 drops of diphenylamine and titrate with the potassium ferrocyanide solution as under 7.2.

7.1.7 Diphenylamine—Melt 1 g diphenylamine in a beaker, cool and make up to 100 ml with concentrated sulphuric acid.

7.1.8 Ammonium Chloride Solution—Dissolve 20 g ammonium chloride with 100 ml water.

7.1.9 Dilute Sulphuric Acid—(1 : 4) and (2 : 98).

7.2 Procedure—Evaporate the combined filtrates reserved under 6.2.1, or the electrolyte reserved under 5.3.1 if cadmium is absent, to about 100 ml and add ammonium hydroxide solution until the precipitate at first formed just fails to re-dissolve. Add 30 ml of formic acid and dilute to

200 ml with water. Heat to boiling and pass a rapid stream of hydrogen sulphide for 15 minutes. Allow to stand for 30 minutes. Filter on a close-texture paper and wash with formic acid wash solution. Dissolve the zinc sulphide on the filter with 50 ml of hot dilute hydrochloric acid catching the solution in the original beaker, and wash the paper thoroughly with hot water. Boil the solution to expel hydrogen sulphide. Neutralize with ammonium-hydroxide and thereafter make the solution just acidic with dilute sulphuric acid (2:98) after putting a small litmus paper. Add 25 ml of dilute sulphuric acid (1:4), just boil and cool to the room temperature. Add 20 ml of 20 percent ammonium chloride and 3 drops of diphenylamine. The volume of the solution should be near about 100 ml and kept in a conical flask. Titrate with standard potassium ferrocyanide solution. The titration should be carried out slowly at the initial stage. During the addition of potassium ferrocyanide the colour of the solution becomes intense blue and before the end point is reached, a sudden change to violet colour will be the indication of the near approach of end point. Shake vigorously and titrate drop-wise until the colour changes to pea-green.

7.3 Calculation

$$\text{Zinc, percent by weight} = 100 \times \frac{(A \times B)}{C}$$

where

A = volume in ml of potassium ferrocyanide solution,

B = weight in g of zinc which is equivalent to 1 ml of potassium ferrocyanide solution, and

C = weight in g of the material taken for the test.

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